Transition-metal silicides formed by ion mixing and by thermal annealing: Which species moves?

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The moving species during the formation of Pt_2Si, Ni_2Si, and $CrSi_2$ by both ion mixing with 300–600 keV Xe ions and thermal annealing is identified with inert markers using backscattering spectrometry. Samples of metal-on-silicon and silicon-on-metal have been used, evaporated on SiO_2 substrates with two very thin markers (Mo for Pt_2Si, W for Ni_2Si and $CrSi_2$) placed at the metal–silicon interface, and at the bottom interface with the SiO_2 substrate. Monitoring the separation of the two markers as a function of the amount of silicide formed determines the ratio of atomic transport through the growing silicide layer. The results establish that the dominant moving species in both silicide formation processes is the same for the refractory metal-silicide $CrSi_2$, e.g., Si, whereas different atomic transport ratios are found in the case of the near-noble metal silicides Pt_2Si and Ni_2Si. This outcome is discussed in terms of high-temperature effects during thermal formation of transition-metal silicides.

I. INTRODUCTION

For a complete understanding of thin-film reactions, the knowledge of the moving atomic species as well as of the atomic transport mechanism involved is essential. Both inert marker and atomic tracer experiments are needed to obtain the required information. For many transition-metal silicides, the dominant moving species is well known for formation by thermal annealing. The same compounds can be formed also by ion irradiation. The crucial question then is whether the dominant moving species is the same for this process as it is for thermal annealing. An identical result would suggest great similarity in the two processes, while dissimilar results would show unequivocally that the two processes differ. In this paper, we study the question of the moving species for the reaction between thin-film bilayers of amorphous evaporated Si and polycrystalline evaporated Pt, Ni, and Cr under thermal annealing, and under ion mixing with Xe ions of 300–600 keV, using inert markers. For ion mixing, the samples are kept at temperatures low enough to prevent thermal reaction but high enough to insure silicide formation under irradiation. In these systems, the compounds formed (Pt_2Si, Ni_2Si, $CrSi_2$) and their growth kinetics are known to be alike for both formation processes, suggesting at least some similarities between them.

The first inert marker experiments on ion mixing have been performed by Hung *et al.*[1,2] on bilayers of Ni/Si, Pd/Si, and Cr/Si using Ar irradiation. They find that for $CrSi_2$, Si is the moving species, as is the case for thermal annealing.[3] For Ni_2Si a ratio for atomic transport of Si to Ni greater than 2 : 1 is reported, whereas thermally Ni moves almost exclusively.[4] In Pd_2Si, Si is found to be the dominant moving species for ion mixing; thermally, both Pd and Si have been reported to move.[5] These results show that the moving species for ion mixing and thermal annealing sometimes concurs (as in $CrSi_2$) and sometimes does not (as in Ni_2Si). The inert nature of a marker can be established only indirectly by confirming that the outcome is independent of the choice of the marker. Hence, our experiments, which use markers and ions different from those of Hung *et al.*, provide such a test for Ni_2Si and $CrSi_2$.

II. EXPERIMENTAL PROCEDURE

Oxidized Si wafers were cleaned ultrasonically in TCE, acetone, and methanol before loading into an oil-free electron-gun evaporation system. Thin films of Si, metal (Pt, Ni, or Cr), and a marker (Mo, W) were evaporated at a pressure below 5×10^{-7} Torr in a single pumpdown. Two types of samples were prepared, as illustrated in Fig. 1. The layer thicknesses were optimized to the projected range of 600-

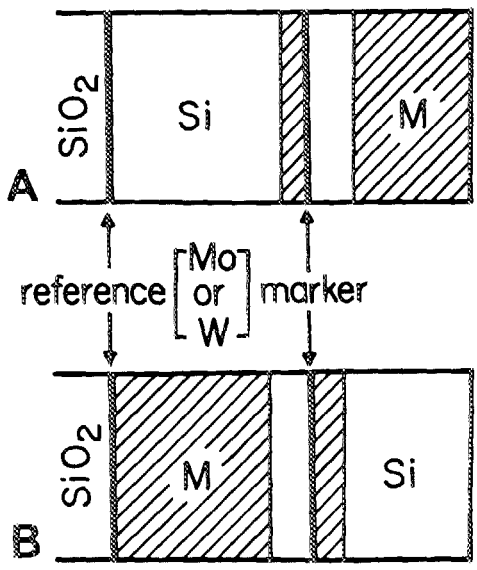

FIG. 1. Two sample configurations (as-deposited) used throughout this study. M: metal (Pt, Ni, or Cr). Layer thicknesses vary from system to system (see text). The bottom layer of Mo (or W) is called "reference marker" in this paper in order to clearly distinguish it from the marker.

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keV Xe to insure penetration of the ions through both interfaces of the growing silicide until completion of the reaction, but at the same time to prevent the reference layer (see Fig. 1) from irradiation-induced broadening (sample A: top layer: 300–500 Å Pt, 500–700 Å Ni, or 550 Å Cr, respectively; bottom layer: 2000 Å Si; *sample B*: top layer: 500–2000 Å Si; bottom layer: 550 Å Pt, 650 Å Ni, or 1000 Å Cr, respectively). The markers used are Mo (8 Å) for Pt_2Si, and W for Ni_2Si (15 Å) and $CrSi_2$ (8 Å). This choice allows easy separation of the backscattering signals from the marker and the metal. The reason for the increased marker thickness in the case of Ni_2Si is a pronounced interference between the marker signal and that from the implanted Xe in the backscattering spectra [see Fig. 2(b)]. Two thin layers of metal and Si are added adjacent to the marker to embed the latter within a homogeneous silicide layer (150 Å Pt_2Si, 150 Å Ni_2Si, 900 Å $CrSi_2$) after a preannealing step. This precaution hinders interfacial drag of the marker during subsequent silicide growth.[6] Ion mixing has been carried out at a pressure below 7×10^{-7} Torr with Xe ions of 300–600 keV and doses up to 3×10^{15} Xe/cm^2 (Pt_2Si), 2×10^{16} Xe/cm^2 (Ni_2Si), and 4×10^{16} Xe/cm^2 ($CrSi_2$). During irradiation, the samples were kept at temperatures high enough to ensure silicide formation under irradiation, but low enough to prevent thermal reaction (100 °C for Pt_2Si, 110 °C for Ni_2Si, 275 °C for $CrSi_2$). That no reaction did occur without irradiation has been verified with samples mounted on the same heated sample holder during ion irradiation, but protected from it by a cover. Thermal annealing has been performed in a vacuum furnace (about 7×10^{-7} Torr) at various temperatures. The compounds formed were checked by x-ray diffraction in a READ camera using Cu$K\alpha$ radiation. The induced reactions for both processes and the associated marker shifts were monitored by 2-MeV $^4He^+$ backscattering spectrometry. Marker shifts are determined relative to the position of the reference layer (see Fig. 1), rather than to the sample surface. This eliminates ambiguities introduced by sputtering effects. Comparison between the outcome of the marker experiment on samples A and on samples B allows to discriminate between a reaction-induced shift of the marker and a possible superimposed movement (see also Sec. IV). In the case of overlapping backscattering signals from the marker and the implanted Xe, spectra from coirradiated, similar samples containing no marker have been used to subtract the interfering Xe signal.

III. RESULTS

Backscattering spectra for the three systems investigated are shown in Fig. 2 after preannealing (solid lines), and after thermal reaction (empty circles), or ion mixing (closed circles). Theoretical levels and their slopes for pure Pt, Ni, or Cr, and for the three silicides Pt_2Si, Ni_2Si, and $CrSi_2$, have been calculated from the Si signal height of the SiO_2 substrate (not shown in the spectra of Fig. 2); these levels are indicated with solid lines in the metal signals.

The upper insert in Fig. 2(a) shows the configuration of a sample with Pt on top after preannealing for 10 min at 250 °C. In this case the layer thicknesses were 2000 Å of *a*-Si, 150 Å of Pt_2Si containing a Mo marker and 250 Å of Pt.

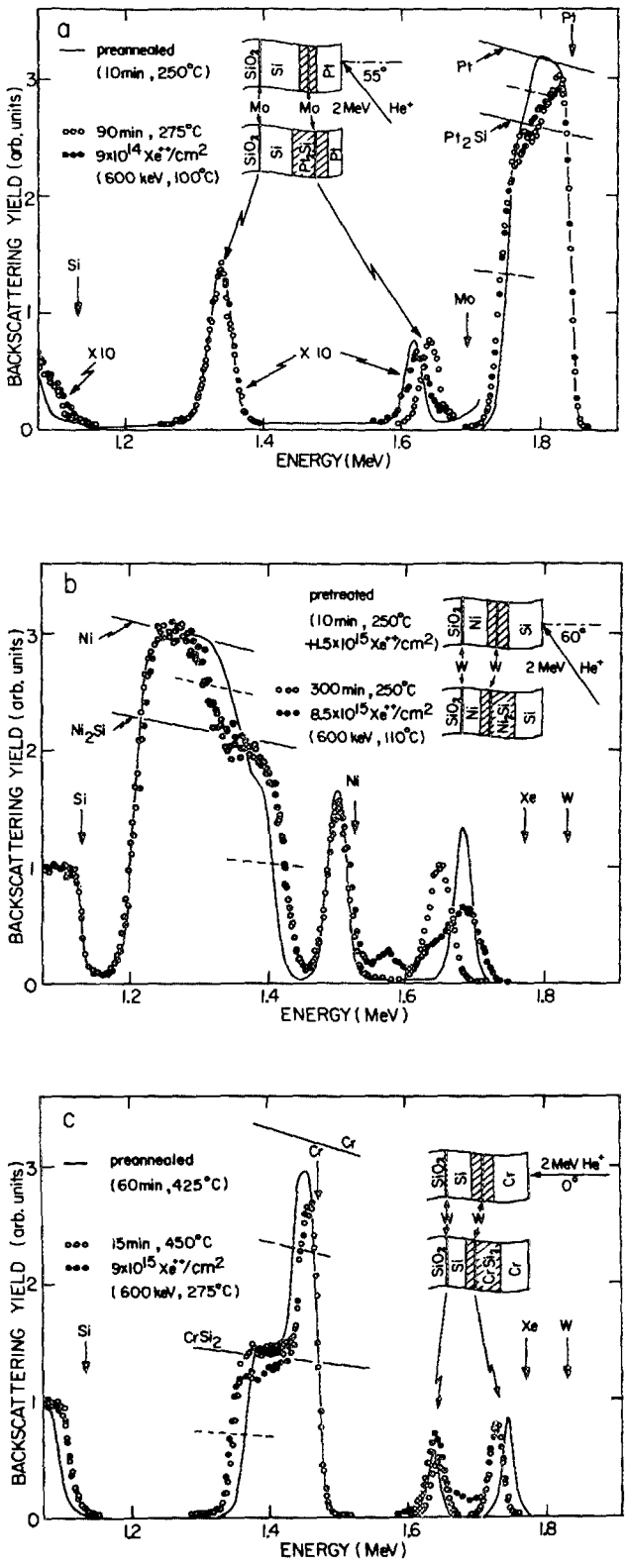


FIG. 2. 2-MeV $^4He^+$ backscattering spectra obtained from preannealed samples of (a) Pt/Si, (b) Si/Ni, and (c) Cr/Si before (solid line spectra) and after thermal annealing (empty circles) as indicated in the inserts, or ion mixing (filled circles) with 600-keV Xe^{+} at indicated temperatures and doses. The particle detector was mounted at 170° with respect to the incident $^4He^+$ beam. Calculated levels for the pure metals and the compounds formed are indicated with solid lines. The formed silicides are measured from energy widths defined by the half heights to these levels (dashed lines). Note the different shifts of the marker signals upon thermal reaction and ion mixing.

Irradiation with 9×10^{14} Xe/cm^2 (filled circles) shifts the marker signal very slightly towards higher energies with respect to the preannealed sample (solid line). This signal also contains a contribution from the implanted Xe, but we know from a comparison with spectra of coirradiated, similar samples without a marker, that this interference is insignificant. The unchanged signals at about 1.35 MeV confirm that the Mo reference marker has not been disturbed by the irradiated ions; they also reveal that sputtering effects are negligible in this case (no shift). The Pt signal indicates that most of the Pt is intermixed with Si. In comparison to the indicated theoretical height for Pt in Pt_2Si, the observed signal reveals that the compound layer produced by ion mixing has an average composition near that of Pt_2Si whereas its actual composition varies with depth. Also included is a spectrum obtained after thermal annealing at 275 °C for 90 min (empty circles). Clearly, the marker shifts towards higher energies much more pronouncedly than for ion mixing, revealing a different atomic transport. Here, again, the Pt signal reflects a slightly nonuniform atomic composition throughout the mixed layer. The mild depression of the Pt signal with respect to the calculated height for Pt_2Si is due to the presence of the Mo marker at this depth. For both samples (the ion mixed and the thermally annealed) x-ray analysis confirms the formation of Pt_2Si.

Figure 2(b) shows similar spectra for the formation of Ni_2Si. In this case, the analysis has been carried out mainly on samples with Si on top because of severe sputtering and surface roughening encountered in ion-mixed samples of the reversed layer sequence. Because of the mentioned need for a thicker marker layer (15 Å instead of 8 Å), the samples have been irradiated with 1.5×10^{15} Xe/cm^2 after preannealing for 10 min at 250 °C in order to disperse the marker and lessen its effect on the subsequent silicide formation process. This improvement was verified from the measured reaction rates which increased from about 30% (without irradiation) to about 50% (with irradiation) of the reaction rates quoted in the literature for thermal annealing of evaporated bilayers of Si and Ni.[7] The sample configuration after this pretreatment is shown in the upper insert of Fig. 2(b). The layer thicknesses are 600 Å of Ni, 280 Å of Ni_2Si containing a W marker, and 1000 Å of *a*-Si. The marker is seen to shift towards the unreacted Ni layer upon thermal annealing, but it remains almost fixed during ion mixing. A distinct broadening of the marker is apparent in the latter case. However, the signal still allows an appropriately accurate determination of its mean position. The low-energy side of the marker signal overlaps with the signal from the implanted Xe. This interference has been subtracted using the spectrum from a similar, coirradiated sample without W marker. The dip in the Xe signal at 1.6 MeV is caused by the marker, located at this depth. The signal from the W reference marker exhibits little change upon reaction except for a small shift to higher energies, corresponding to the amount of Si lost by sputtering during ion mixing. Again, the step in the metal signal reveals the formation of Ni_2Si (confirmed by x-ray analysis) with some composition variations throughout this silicide layer. Note that the step height is distinctly below the calculated height for Ni_2Si (solid line) in the pretreated sample spectrum. This is caused by the high W concentration in this region.

Backscattering spectra for the formation of $CrSi_2$ are shown in Fig. 2(c). Here, the preannealing step indicated in the insert yielded layer thicknesses of 1500 Å of *a*-Si, 900 Å of $CrSi_2$ with an embedded W marker, and 300 Å of Cr. Annealing at 450 °C and ion mixing with 600-keV Xe at 275 °C can be seen to produce very nearly the same marker shifts, in contrast to the cases of Pt_2Si and Ni_2Si. The W reference signal appears at slightly higher energies in the spectrum of the thermally annealed sample (empty circles) compared to the two other spectra. This represents thickness variations from sample to sample rather than a real shift. Such variations do not affect our results, however, since all samples have been measured individually before and after reaction. For this system, the formed silicide appears to be homogeneous in composition for both thermal annealing and ion mixing (Cr signal). Ion mixing, however, produces a Si-rich silicide, as evidenced by the low step height (Cr in $CrSi_2$). Nevertheless, x-ray analysis proves that $CrSi_2$ forms, the diffraction rings being identical for both processes.

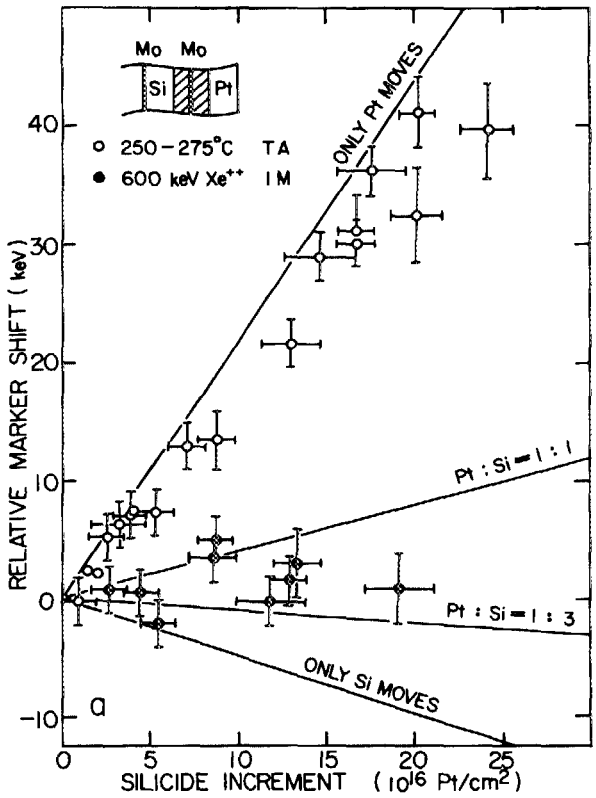
Marker shifts have been measured from backscattering spectra with respect to the position of the signal from the reference marker. They have been corrected for the shift produced by the implanted Xe; the He added during backscattering has negligible effect on the separation of the two markers. Since different incident angles have been used throughout the backscattering experiments, all shifts have been converted to normal incidence to get comparable data.

A. Pt_2Si

Figure 3 gives these marker shifts for the formation of Pt_2Si as a function of the silicide increment, i.e., the number of Pt atoms per unit area added to the silicide layer. This number is obtained from the increase in the energy width of the Pt signal in Pt_2Si. That width is defined by the half heights [dashed lines in Fig. 2(a) of the steps in the Pt signal corresponding to the interfaces Si/Pt_2Si and Pt_2Si/Pt. Figure 3(a) shows the data for the samples with Pt on top, whereas the results for samples of interchanged layer sequence are given in Fig. 3(b). The full circles are the data for ion mixing; a dose of 1×10^{15} Xe/cm^2 leads to a silicide increment of about 10^{17} Pt/cm^2. The empty circles are the results for thermal annealing. The solid lines in Fig. 3 correspond to marker motions calculated using the surface energy approximation for several indicated ratios of relative contributions of the two species to the atomic transport. The thermal annealing data clearly indicate a very predominant motion of Pt. When a straight line is least squares fitted to the actual data points, we find for both layer sequences an atomic transport ratio of about 13 : 1 in favor of Pt. In contrast to that, ion mixing data reveal that both species are involved in the transport with a ratio of 1 : 1 to 1 : 3 in favor of Si. This shows that the two processes differ for the formation of Pt_2Si.

B. Ni_2Si

A similar plot of the marker shift as a function of silicide increment is presented in Fig. 4 for Ni_2Si, again for samples



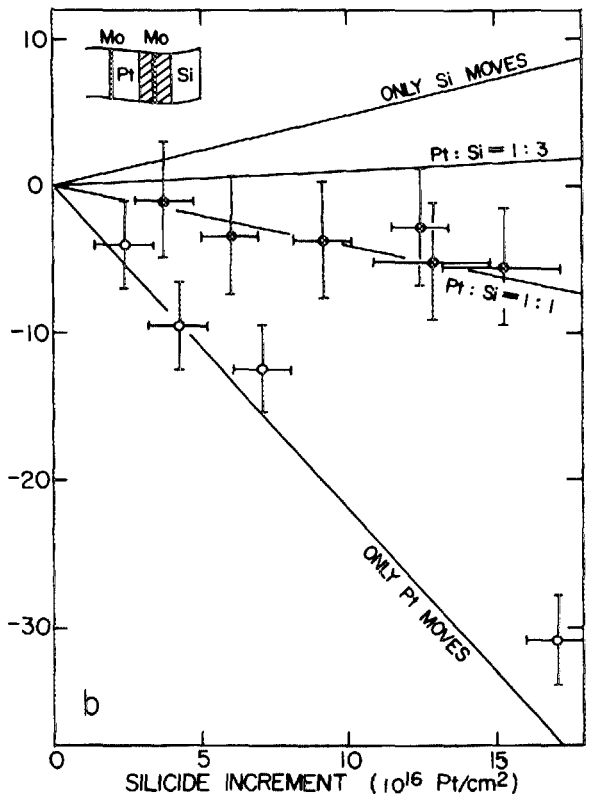


FIG. 3. Marker shifts, measured from spectra as shown in Fig. 2 with respect of the Mo-reference signal and converted to normal incidence, as a function of silicide increment for bilayers of (a) Pt on Si and (b) Si on Pt. Empty circles are for thermal annealing at indicated temperatures. Closed circles are for ion mixing with 600-keV Xe^{++}, with the samples kept at 100 °C during irradiation. Solid lines represent theoretical marker shifts for various indicated atomic transport ratios.

with either metal [Fig. 4(a)] or Si [Fig. 4(b)] on top. The thermal annealing data (empty circles) convincingly reveal pure Ni movement for both layer sequences, whereas both atomic species move during ion mixing (filled circles), as is the case for Pt_2Si. In this case, a dose of 1×10^{16} Xe/cm^2 is needed to increase the silicide thickness by 10^{17} Ni/cm^2. The higher doses needed for Ni compared to Pt are consistent with reaction rates for ion mixing, reported to be much lower for Ni/Si bilayers than for Pt/Si bilayers.[8] Data for 300-keV Xe irradiation are also included in Fig. 4 (triangles); at this energy, the Xe ions only cross the first interface of the silicide. In this case, backscattering spectrometry suggests atomic compositions of the mixed layer between Ni_2Si and NiSi. Therefore, marker shifts calculated for the formation of NiSi have also been included in Fig. 4 (dashed lines). Additional work is needed to decide whether or not this compound forms during 300-keV Xe irradiation, since x-ray analysis could not corroborate such an assumption. Note, however, that such composition variations do not significantly change the calculated marker shifts. The sputtering process mentioned previously for samples with Ni on top, combined with a substantial marker broadening [see also Fig. 2(b)] leads to the pronounced scattering and considerable uncertainty of the ion mixing data in Fig. 4(a). Yet this uncertainty does not affect the outcome of the marker experiment which, again, reveals different atomic transport for the two silicide formation processes.

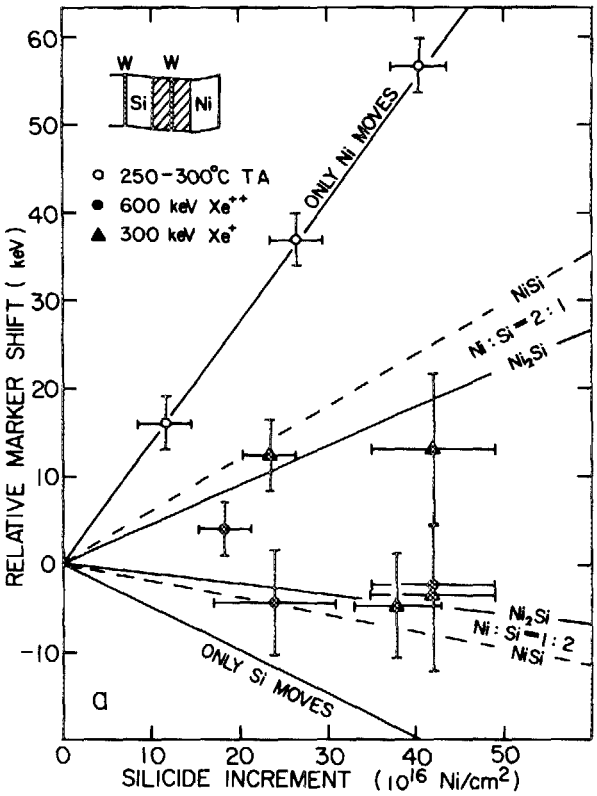
C. $CrSi_2$

Marker shifts for the formation of $CrSi_2$ are plotted in Fig. 5 as a function of the number of Si atoms added to the silicide layer. In this plot a silicide increment of 10^{17} Si/cm^2 corresponds to a dose of about 5×10^{15} Xe/cm^2 for 600-keV Xe (filled circles). Results for 300-keV Xe irradiation are also included (triangles). At this energy, only the Cr/$CrSi_2$ interface is irradiated, but this is known to be sufficient for the growth of $CrSi_2$.[9] The slopes to the data for both layer sequences and both ion energies unequivocally establish that Si is the only moving species during both temperature-induced and irradiation-induced formation of $CrSi_2$. Figure 5(b) suggests an initial delay in the marker shift (dashed line). We have established that this delay is caused by an incomplete consumption of the interposed Si and Cr layers adjacent to the W marker (see Fig. 1) after the preannealing step. Up to about 5×10^{16} Si/cm^2, the silicide thus grows without the need of any atomic transport across the plane defined by the marker.

IV. DISCUSSION

A. Pt_2Si

It is commonly believed that Pt is the only moving species for the formation of Pt_2Si by thermal reaction of a Pt film with a Si substrate. If that is correct, the systematic deviation we observe for the thermal annealing data, particularly in Fig. 3(a), could be attributed either to the amorphous nature of the Si film or to an interference produced by the marker. Such an interference is suggested by the reduced reaction rates we measure for samples with a Mo marker,



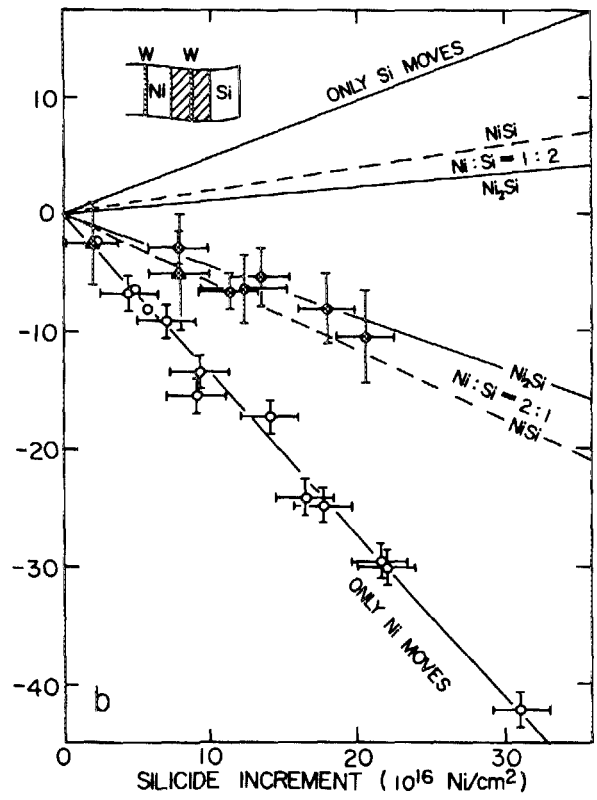


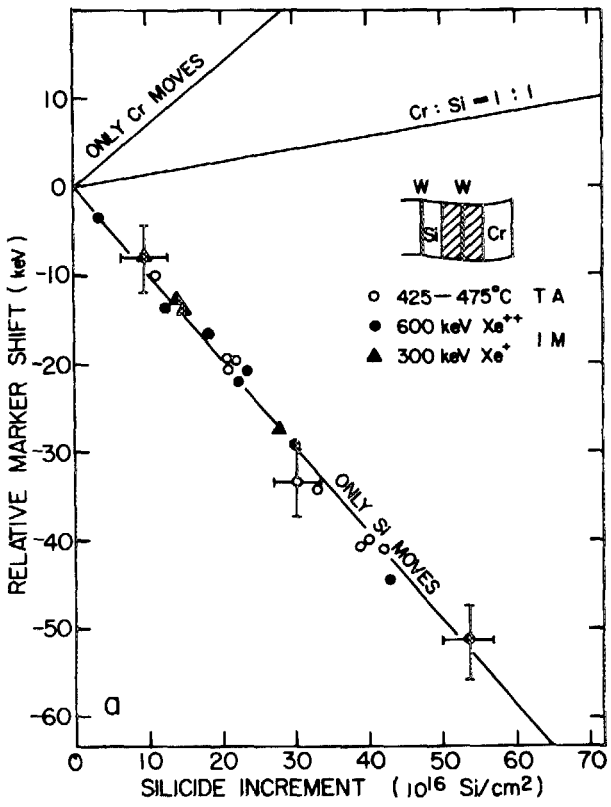
FIG. 4. As Fig. 3, except for Ni/Si bilayers. Ion mixing was carried out at 110 °C. Triangles are for ion mixing with 300-keV Xe^+ irradiation.

these rates being only about 4%[10] to 35% [11] of those quoted in the literature, as well as by the composition variations discussed in connection with Fig. 2(a). A literature survey of inert marker experiments carried out on the near-noble metal silicides reveals, however, that only one such experiment is reported for Pt_2Si.[12] That paper states that both Si and Pt are involved in the transport, but no ratio is given, and no actual data are provided on the kinetics, the marker used, and the shifts observed, since the main issue of that paper is not the identification of the dominant moving species. It could therefore be argued also that, in contrast to common belief, both species move during thermal annealing in the absence of a marker (as suggested in Ref. 12), and that it is the presence of the Mo marker which suppresses Si motion in our experiment. We note, however, that the marker does not change the compound formed (Pt_2Si). Also, the low reaction rate measured for our samples containing a marker is not surprising in view of the known high sensitivity of the rate of thermal formation of Pt_2Si to impurities.[13] In the case of Ni considered below, there is no such ambiguity; Ni is quite clearly the dominant moving species in the thermal formation of Ni_2Si.[14] Because of the general similarity that exists between the thermal reactions of Ni and Pt films with Si, including such special effects as the redistribution of impurities during the reaction,[15–17] we believe that Pt is the moving species in the thermal formation of Pt_2Si in the absence of a marker. Our subsequent discussions shall therefore proceed from that assumption. We observe, however, that an independent study of the dominant moving species in the formation of Pt_2Si by thermal annealing should be undertaken with a marker other than Mo that reduces the reaction rates little. That study should include quantitative information as is provided here for the first time for this system.

As for the ion-mixing results, it is clear that both species are involved in the transport during formation of Pt_2Si. The actual data show a slight difference for samples with different film sequences [Figs. 3(a) and 3(b)]. A possible explanation for this difference is to assume an irradiation-induced shift of the marker away from the sample surface, superimposed on the shift produced by the reaction-related atomic transport. That additional shift, if it is real, amounts to about 1.2 keV/10^{17} Pt cm^{-2}, or about 10 Å for a dose of 1×10^{15} Xe/cm^2. In fact, an inward shift of this magnitude has been reported for a Pt marker in *a*-Si upon irradiation with Xe ions.[18] If such an interpretation of our results is true, the actual ratio of atomic transport during ion mixing turns out to be about 2 : 3 in favor of Si.

B. Ni_2Si

Because of the similarities between Pt and Ni in a thermal reaction with Si, it is interesting to compare the outcome of the marker experiments presented here for the two systems Pt/Si and Ni/Si. Figure 4 showed pure Ni motion during thermal annealing, in agreement with the literature.[4] As is the case for Pt_2Si, both species move during ion mixing. Again, the apparent ratio of the atomic transport for Ni and Si differs somewhat for the two sample configurations [Figs. 4(a) and 4(b)]. Here, this difference suggests a superimposed-marker shift away from the sample surface of about 2 keV/



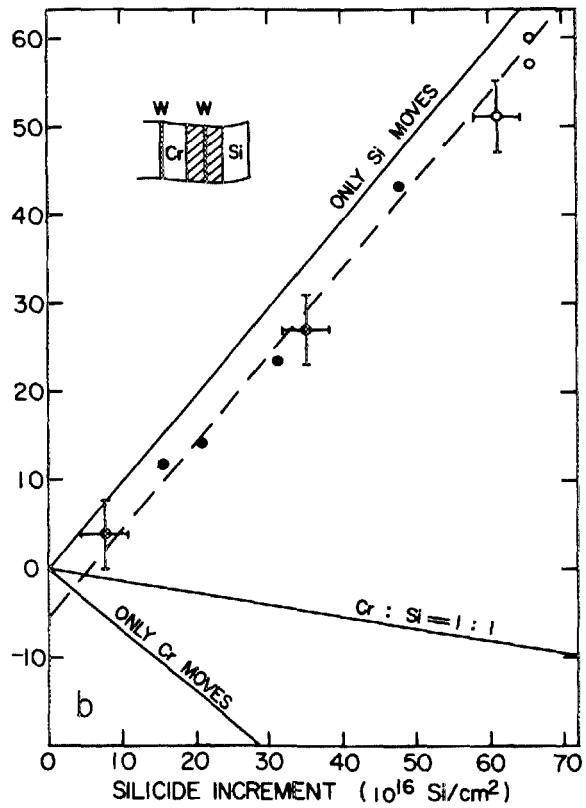


FIG. 5. As Fig. 4, except for Cr/Si bilayers. Ion mixing was carried out at 275 °C. Dashed line: slope to data; initial delay of the marker shift is caused by incomplete preannealing step (see text).

10^{17} Ni cm^{-2}, or about 18Å for a dose of 1×10^{16} Xe/cm^2. The atomic transport ratio thus appears to be about 3 : 2 in favor of Ni. Although this ratio is different from the ratio of 2 : 3 found for Pt_2Si both ratios are near 1 : 1.

Another inert marker experiment has been performed recently on beam-induced Ni_2Si formation.[2] This experiment was performed with Ag as a marker and Ar irradiation. The results concur with ours, although the transport ratio is quoted there as being "greater than 2 : 1" in favor of Si. This difference does not materially affect the conclusion that ion mixing does not reproduce the atomic transport mechanism of a thermal annealing experiment in this system. It thus appears that Ni and Pt behave similarly with respect to the moving species during ion mixing. They differ in similar ways from their behavior upon thermal annealing.

C. $CrSi_2$

The thermal reaction of Cr films with a Si substrate departs essentially from the way Ni and Pt react with the same substrate: no metal-rich silicide form, and only $CrSi_2$ is observed, which is the compound in equilibrium with excess Si; the growth is linear in time, rather than parabolic[19]; Si is the moving species, rather than the metal.[3] Our experiments confirm that Si is the only moving species in a thermal formation of $CrSi_2$. In addition we obtain the same result for ion mixing: Cr is not involved in the atomic transport. That outcome, again, concurs with the results reported in Ref. 2.

V. CONCLUSION

Two conclusions follow from the agreement between our results and those by Hung *et al.*[1,2] for Ni_2Si and $CrSi_2$: (i) W and Ag are most probably good inert markers in those compounds, (ii) the atomic transport mechanisms during thermal annealing and during ion mixing differ for Ni_2Si whereas the same species moves for both processes in $CrSi_2$. These conclusions remain valid irrespective of the choice of the ion (Ar or Xe). In addition, we find that Pt_2Si behaves like Ni_2Si.

A bilayer experiment with a film of W on top of a film of Mo on a Si substrate has also been carried out for thermal annealing[20] and for ion mixing.[21] To the extent that Mo may be viewed as an isotope of W, these are also marker experiments. The authors find that for both thermal annealing and ion mixing, only Si moves during compound formation. Hence, in view of this experiment and our results, the commonly applied subdivision of transition metals to characterize silicide formation under thermal annealing seems to be meaningful for ion mixing as well. We would then predict that under ion mixing, Si is the moving species for the refractory transition-metal disilicides, whereas both Si and metal move for the near-noble dimetal silicides. That prediction would be worth testing. This then means that for refractory transition-metal disilicides the dominant atomic transport processes in ion mixing and thermal annealing are probably similar, but that they are not for near-noble dimetal silicides. What these transport processes in fact are would of course be very interesting to know. However, inert marker experiments only provide the net balance of atomic motion

through a plane defined by the marker, and by themselves do not characterize the transport processes involved in the reaction. Without additional information, as provided, for example, by tracer experiments, the nature of the transport process must remain a matter of conjecturing. Doing so, we note that the motion of Si appears to always be involved in silicide formation by ion mixing. We further note that during thermal formation of Ni silicides, Si also moves when the annealing is performed at elevated temperatures[22] and that elevated temperatures are always required for thermal growth of refractory metal disilicides; there, Si always moves. The inference then is that the Si motion during ion mixing is due to a process that is also activated at elevated temperatures during thermal annealing. The same idea has been expressed by Hung *et al.*[2] in terms of the particular process of Si vacancy formation. Moreover, high temperatures have also been invoked to model ion mixing of metal/Si bilayers by a thermal spike[23] and to explain systematic trends in ion mixing of metallic bilayers.[24]

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